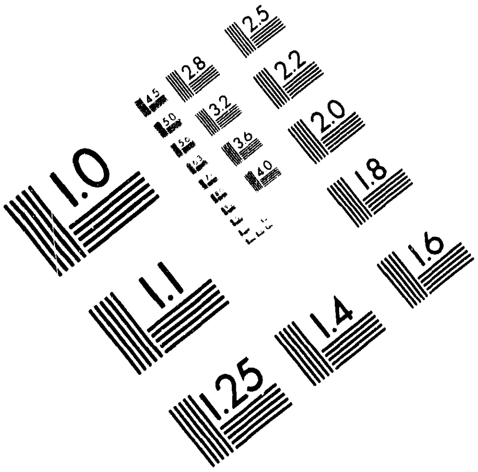

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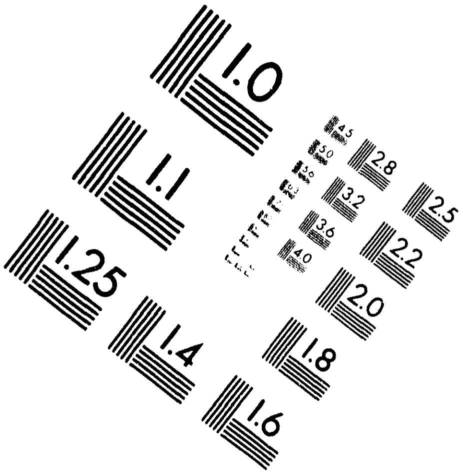

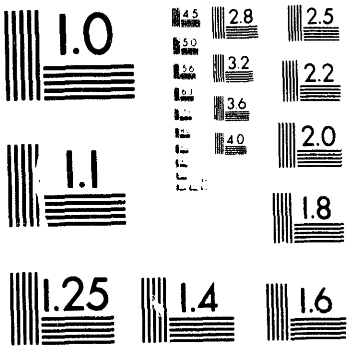

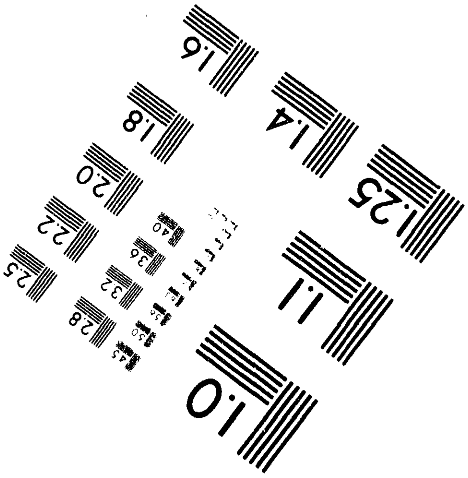

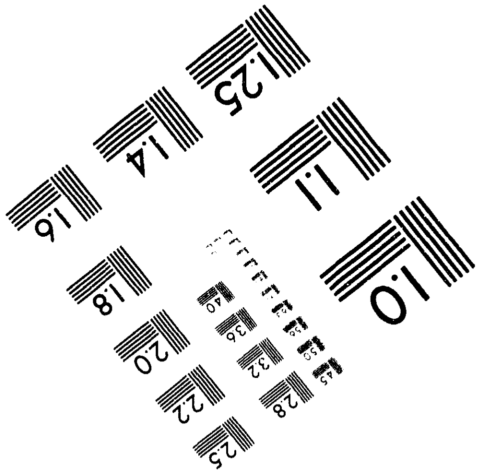

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FABRICATION AND PROPERTIES OF ALUMINA MATRIX COMPOSITES CONTAINING NICKEL ALUMINIDE REINFORCEMENTS

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Abstract

Ductile nickel-aluminide intermetallic alloys have been successfully used to toughen ceramic materials intended for use at a wide range of temperatures. Traditional ceramic processing procedures have been used to produce a variety of microstructures. The fracture toughness increases with increasing particle aspect ratio, however, the flexural strength decreases with increasing particle size. Fracture toughnesses up to 7.6 MPa $m^{1/2}$ and flexural strengths up to 550 MPa were observed in an alumina composite containing 10 vol.% nickel aluminide.

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Introduction

The incorporation of ductile phases into glass and ceramic matrices can result in significant increases in the fracture toughness of the composite.(1-3) The toughening occurs through the plastic deformation of the metallic particles in the crack tip wake region. To accomplish this, the particles must bridge the crack, and the dissipation of strain energy by the deformation of bridging particles will result in an increase in the fracture toughness of the composite, as compared to the monolithic ceramic. The toughness enhancement will depend on the debonding properties of the metal/ceramic interface, the flow properties of the metal, and the particle cross-section bridging the crack. Many of the alumina-based systems recently investigated utilize either nickel (4) or aluminum (3,5) reinforcements, both of which will exhibit decreased yield strength, and thus toughening behavior, with increasing temperature. Recently developed high-temperature Ni_3Al-based intermetallic alloys, which exhibit ductilities exceeding 40%(6), have been successfully incorporated into carbide and oxide matrices(7). The WC/Ni_3Al composites exhibit increased hardness compared to conventional WC/Co-based hardmetals. Ductile polycrystalline Ni_3Al alloys have the unique property of increasing yield strength with increasing temperature, up to approximately 973-1073K. Single crystals of nickel aluminide are also ductile. In addition, the yield, flow, and hardness of the nickel aluminide alloys can be altered by alloying(8). These properties suggest that the addition of nickel aluminide to ceramic matrices will result in ductile-phase toughening, with the advantage that the properties could be retained at intermediate temperatures. In this investigation, simple powder processing routes were to fabricate alumina/nickel-aluminide composites with variations in microstructure. The effects of particle shape, orientation, and size on the fracture toughness and flexural strength are explored.

Experimental Procedures

Conventional ceramic powder processing routes were used to fabricate alumina composites containing 10 vol. % nickel-aluminide. The use of powder processing allows flexibility to rapidly examine the role of particle size, morphology, and composition on the resultant properties. Alumina (0.5 μm- Sumitomo Corp. or 3 μm - Alcoa Corp.) and nickel aluminide powders (IC50 Ni_3Al - Homogeneous Metals Corp.) were mixed either by: (1) ball-milling for 8-24 h, in hexane, using either alumina or WC media, or (2) attrition-milling for 2-4 h, in isopropanol, with either alumina or yttria-stabilized zirconia media (Y-TZP). The use of hexane, which is readily volatilized, as the solvent prevented the heavier nickel aluminide particles from settling during drying. Powders processed using isopropanol were dried on a hot-plate with continuous stirring to prevent settling of the nickel aluminide particles. The nickel aluminide powder used was sized by ultrasonically sieving -325 mesh powders into the size classes: -45/+38 μm, -38μm/+15 μm, and - 15 μm. Milling with higher density (WC or Y-TZP) ball-milling media, rather than alumina media, resulted in flattened, penny-shaped nickel aluminide particles. This choice of milling media thus allows us to manipulate the nickel aluminide particle shapes in an easy and reproducible manner. The homogeneity of the milled powders could readily be checked under an optical microscope prior to hot-pressing. Specimens were hot-pressed, in an argon atmosphere, at temperatures ranging from 1623-1823K with applied pressures from 14-35 MPa for 90 min. Nickel-aluminide wires (IC50)0.38 mm in diameter were also incorporated into alumina by hot-pressing at 1623K for 90 min at 35 MPa. Densities were determined by Archimedes' principle. Flexural strengths and fracture toughness values were obtained in four-point bending (bar dimensions approx. 2.54 x 3 mm) with the loading points located at inner and outer spans of 6.35 and 19.95 mm respectively. Indentation loads of 20 kg were used and the fracture toughnesses were determined using the procedures of Cook and Lawn (9). In addition, fracture toughness values were determined with applied-moment double cantilever beam specimens (AMDCB).

Results and Discussion

In carbide- and nitride-based materials, the wetting angles for nickel aluminide on ceramic substrates are low (e.g 15° for TiC), therefore composites can be consolidated via liquid-phase-sintering. For alumina substrates, however, the wetting angles varied from 76° to > 90°, depending on the alloy composition, as shown in Table I. Therefore, in order to consolidate alumina/nickel-aluminide composites, densification must occur via sintering of the alumina matrix. The fine alumina powder (0.5 µm) could be fully densified by hot-pressing at 1623K with 35 MPa applied pressure. The coarse alumina powder, however, could not be densified under these conditions. Since stoichiometric nickel aluminide melts at 1663K, significant nickel aluminide was exuded from the composite prior to densification of the 3-µm-sized alumina.

Table I: Results of 1450°C wetting experiments on Alumina (AD995) in vacuum.

Composition, at. %	Description	Wetting Angle (Degrees)
Ni-16Al-8Cr-1Zr-0.1C-0.1B	adherent	80
Ni-22Al-1Ti-0.1B-0.1C	broke off	80
Ni-18Al-5Ti-0.1B-0.1C	broke off	79
Ni-22Al-1Y-0.1B-0.1C	broke off	>90
Ni - 22 Al - 1 Zr - 0.1B	adherent	>90
Ni - 16 Al - 8 Cr - 1 Zr - 0.1 B	broke off	>90
Ni - 23 Al - 1C - 0.1 B	broke off	76
Ni - 22 Al - 1 Zr - 1 C - 0.1 B	broke off	78
" "	adherent	77
Ni - 22 Al - 1 Zr - 0.1 C -0.1 B	adherent	76
" "	adherent	77
Ni - 18 Al - 5 Zr - 0.1B	broke off	-
Ni - 22 Al - 1 Y - 0.1B	broke off	>90

The microstructures of fully dense alumina-based composites fabricated from the 0.5 µm alumina under a variety of processing conditions are shown in Figure 1. High-energy milling with dense milling media does not comminute the Ni_3Al particles, Figure 1(b) and 1(c), but simply flattens the powder particles into platelets. Note that the particles tend to be aligned with the broad faces perpendicular to the hot-pressing direction. The fracture toughnesses of these composites, as determined by indent-and-fracture bend tests with the crack plane parallel to the hot-pressing axis, are inset in Figure 1. Note that for a given starting particle size, the fracture toughness increases with increasing aspect ratio, Figures 1(a)-(c). Fracture surfaces of these composites typically exhibit two distinctive fracture modes, as shown in Figure 2. Particles oriented with the crack plane nearly perpendicular to the broad face of the platelet exhibit debonding around the particle perimeter, crystallographic slip features, and necking on the Ni_3Al fracture surfaces, as shown in Figure 2(b). Rounder Ni_3Al particles, and those not oriented with the crack plane nearly perpendicular to the broad face of the platelet, show no particle deformation. Figure 2(c). The flexural strengths of these composites are also inset in Figure 1, and varies primarily as a function of platelet diameter. Presumably, the platelets can act as flaw-initiators, in which case the larger platelets will create larger flaws, resulting in lower flexural strength.

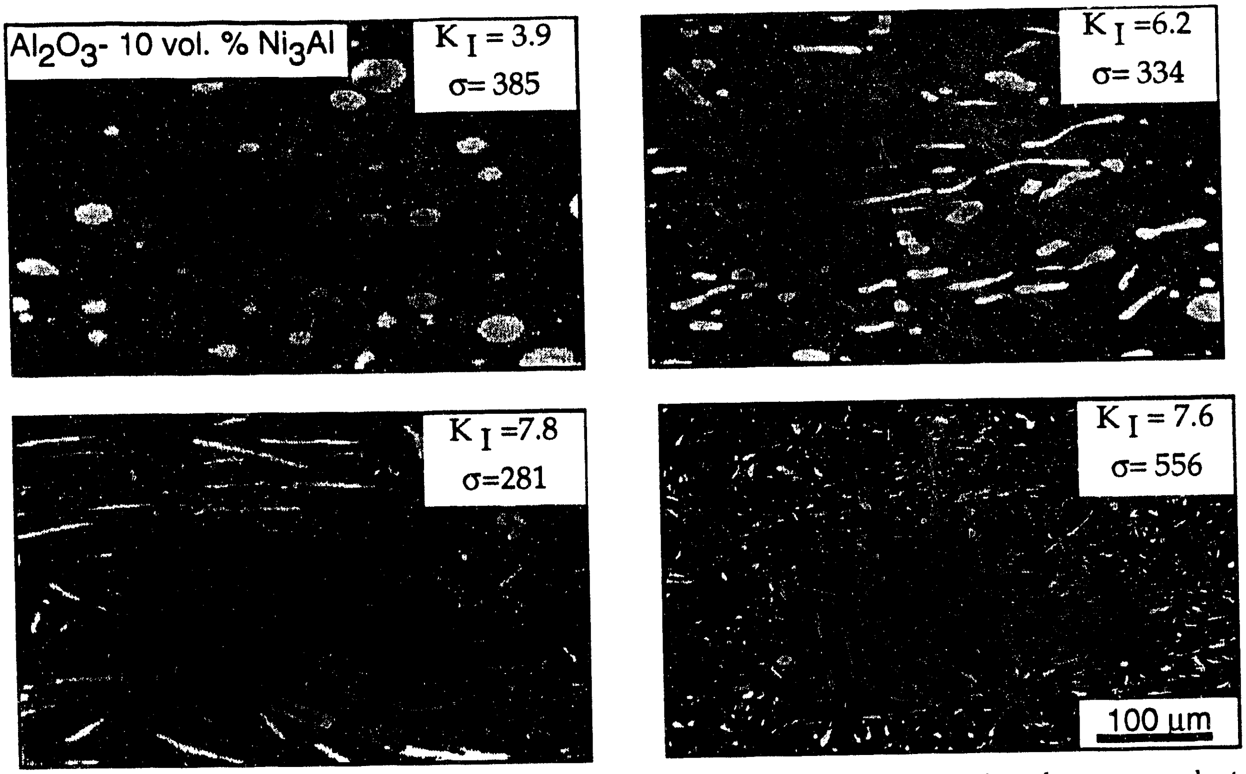

Figure 1 - Optical micrographs of alumina/nickel-aluminide composites hot-pressed at 1623K/35 MPa/90 min. The hot-pressing direction is vertical in each case. (a) -325 mesh Ni_3Al, ball-milled with alumina media, (b) -325 mesh Ni_3Al, ball-milled with WC media, (c) -325 mesh Ni_3Al, attrition-milled with Y-TZP, and (d) -38 µm, +15 µm Ni_3Al, ball-milled with WC media. Relevant fracture toughness (K_I- MPa $m^{1/2}$) and flexural data (σ – MPa) is inset.

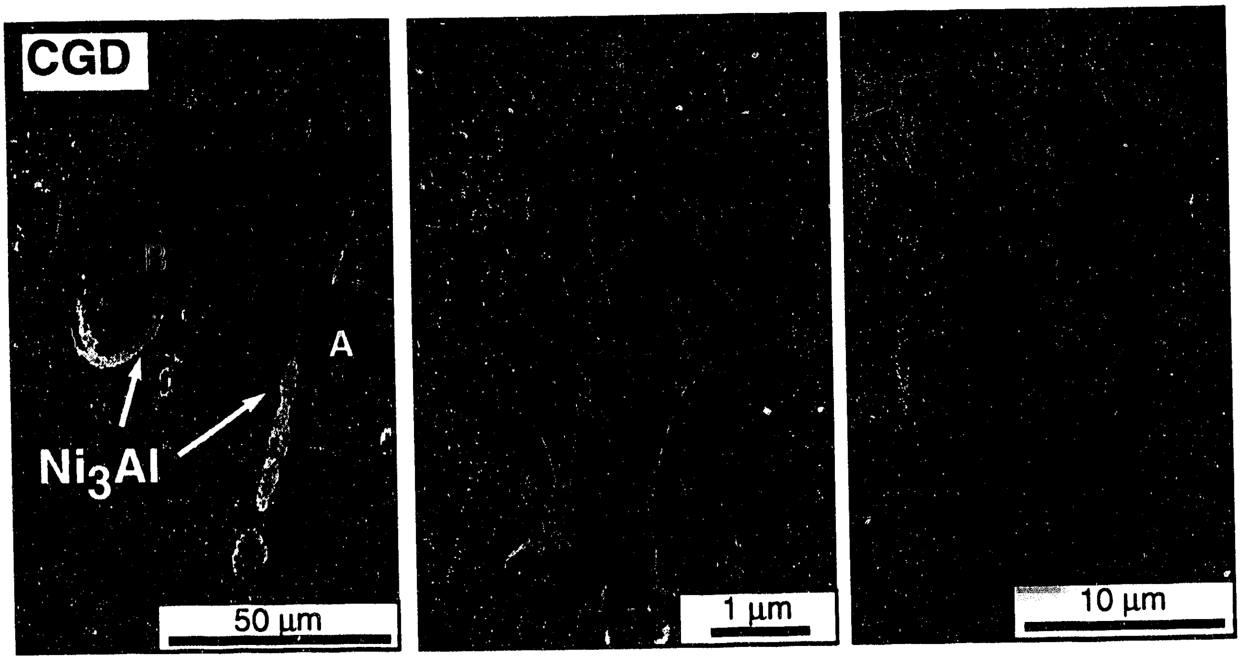

Figure 2 - Fracture surface from microstructure shown in Figure 1(b). The particles exhibit two distinctive fracture morphologies, marked A and B in (a). Particles represented by those marked "A" typically exhibit interfacial debonding and particle deformation as shown in (b). Particles represented by those marked "B" typically do not exhibit particle deformation as shown in (c).

Fracture toughness results from AMDCB tests confirm the importance of the crack plane orientation in these materials. For crack growth with the crack plane oriented perpendicular to the broad face of the platelets, the crack resistance increases with crack length (R-curve behavior) , indicative of crack-bridging effects in the crack wake. When the crack plane is parallel to the broad face of the platelets, however, very modest R-curve behavior is observed.

In order for crack bridging and particle deformation to occur, some debonding is desirable to relieve the hydrostatic constraint of the particle. However, if debonding occurs too readily, the crack will simply follow the particle/matrix interface around the particle, and no bridging will occur. The wetting experiments indicated that there is little chemical bonding occuring, and that the interface is not a strong one. In addition, due to differences in thermal expansion mismatch, the interface is in tension after cooling from the hot-pressing temperature. Therefore, any cracks encountering round particles or elongated particles lying in or near the crack plane can simply follow the interface around the particles once debonding initiates. In this case, negligible particle bridging occurs and the toughness enhancement due to the particles is minimal. However, when irregular or elongated particles are restrained by the matrix (e.g. the particle is favorably oriented with respect to the crack plane or surface roughness locks it into the matrix), bridging can occur. Despite the fact that only the appropriately shaped and oriented Ni_3Al particles are bridging the crack and deforming in the crack tip wake (Figure 2), significant toughness increases can be observed. The potential toughening effects available in this system can be illustrated by examining the effect of ideally-oriented, infinitely-long Ni_3Al reinforcements in alumina. To simulate this idealized microstructure, a composite containing a series of parallel Ni_3Al wires was fabricated. For AMDCB crack growth perpendicular to the wire axes, the toughness increased from 2.5 MPa $m^{1/2}$ (alumina) to 7.5 MPa $m^{1/2}$ in a composite containing the equivalent of only 1 vol. % Ni_3Al in alumina.

Conclusions

Uniform and dense composites of alumina reinforced with ductile nickel aluminide alloys have been fabricated with variations in microstructure. Ductile intermetallic particles have been shown to be effective ductile-phase-toughening additions to ceramic matrices, and have the potential for intermediate temperature performance. Significant increases in the fracture toughness have been observed, up to 7.6 MPa $m^{1/2}$, while maintaining flexural strengths over 500 MPa. This fracture toughness represents a threefold increase over the monolithic fracture toughness of 2.5 MPa $m^{1/2}$. The fracture toughness increases with the particle aspect ratio, with only elongated, favorably-oriented particles deforming in the crack-tip wake.

Although the composites examined in this study were anisotropic, the potential for ductile-phase toughening of ceramics by high temperature intermetallic alloys has been demonstrated. The microstructural variations employed allowed the important parameters for toughening to be identified. The observations of this study also provide some important guidance for the optimized microstructural design of these composites. Since the interface is in tension, and readily debonds, only rough particles, or particles with extended embedded lengths, will enable crack bridging and subsequent particle deformation to occur. Therefore, the ideal microstructure would consist of meandering, interconnected Ni_3Al regions. This microstructure should provide maximum toughness and maintain reasonable flexure properties. However, the lack of sufficient wetting behavior in the alumina/nickel-aluminide systems hinders the fabrication of this ideal microstructure via simple sintering and hot-pressing routes. Since the effectiveness of ductile intermetallic alloys as toughening agents for ceramics has been demonstrated, further studies are underway to fabricate more optimum interconnected microstructures.

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